

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1686b

Nitric Oxide in Nitrogen

(Nominal Amount-of-Substance Fraction - 500 µmol/mol)

This Standard Reference Material (SRM) is a primary gas mixture to which the amount-of-substance fraction, expressed as concentration [1], of secondary working standards may be related. The SRM is intended for the calibration of instruments used for nitric oxide determinations, for monitoring source emissions, and for other applications including the analysis of chemical and combustion process streams.

This SRM mixture is supplied in a DOT 3AL specification aluminum (6061 alloy) cylinder with a water volume of 6 L. Mixtures are shipped with a nominal pressure exceeding 12.4 MPa (1800 psi) which provides the user with 0.73 m³ (25.3 ft³) of useable mixture. The cylinder is the property of the purchaser and is equipped with a CGA-660 stainless steel valve, which is the recommended outlet for this nitric oxide mixture. NIST recommends that this cylinder not be used below 0.7 MPa (100 psi).

Certified Values: This SRM mixture has been certified for nitric oxide (NO) and total oxides of nitrogen (NO_x) concentrations. The certified values, given below, apply to the identified cylinder and NIST sample number.

Nitric Oxide (NO) Concentration = (± 2.5) μ mol/mol Total Oxides of Nitrogen (NO_x) Concentration = (± 2.5) μ mol/mol

Cylinder Number:

NIST Sample Number:

The uncertainties of the certified values include the estimated uncertainties of the gravimetrically prepared primary standards, the imprecision of measurements intercomparing the primary standards to the lot standard (LS), and the imprecision of intercomparing the LS with each of the mixtures comprising the lot. These uncertainties are expressed as expanded uncertainties, $U = ku_c$, with u_c determined from experimental standard deviations and the coverage factor k is equal to 2. Since the concentration values of gaseous SRMs are assumed to be normally distributed with an experimental standard deviation of u_c , the true values for the nitric oxide concentration and total oxides of nitrogen concentration are asserted to lie in the interval defined by the certified values $\pm U$ with a level of confidence of approximately 95 % [2].

Expiration of Certification: This certification is valid until 1 October 2005 within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification will be nullified if the SRM is contaminated or modified.

Cylinder and Gas Handling Information: NIST recommends the use of a high purity, stainless steel, two-stage pressure regulator with a stainless steel diaphragm and CGA-660 outlet to safely reduce the pressure and to deliver this SRM mixture to the instrument. The regulator should be evacuated and purged several times to prevent accidental contamination of the sample. Contamination of this mixture with trace quantities of oxygen will cause a slow decrease in the nitric oxide value.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by J.C. Colbert.

Willie E. May, Chief Analytical Chemistry Division

Gaithersburg, MD 20899 Certificate Issue Date: 30 December 1999

Thomas E. Gills, Director Office of Measurement Services

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The analytical measurements leading to the certification of this current SRM lot were performed by M.E. Kelley of the NIST Analytical Chemistry Division.

The overall direction and coordination of the technical work required for certification of this SRM was performed by F.R. Guenther of the NIST Analytical Chemistry Division.

Mixture Preparation: The gas mixtures comprising this SRM lot were prepared in accordance with NIST technical specifications by a commercial specialty gas vendor under contract to NIST. The specifications stipulate that each SRM mixture be identical in nitric oxide concentration and stable with time.

Analytical Methods: Analysis of the nitric oxide (NO) and the total oxides of nitrogen (NO_x) concentrations were conducted by intercomparing each cylinder mixture to a representative cylinder chosen from the lot, the lot standard (LS), using a chemiluminescent continuous analyzer. Assignment of the NO and NO_x concentrations to the lot was accomplished by intercomparison to primary gravimetric standards using chemiluminescence.

Homogeneity Analysis: Each of the nitric oxide mixtures which comprise this SRM lot was intercompared to the LS at least three times. An analysis of variance indicated that sample to sample nitric oxide concentration differences were statistically significant. This indicates that within the precision of the NIST measurements, all of the cylinders comprising this SRM lot have slightly different nitric oxide concentrations.

Nitric Oxide Concentration Value Assignment: The certified nitric oxide concentrations for this SRM lot were computed from the assigned concentration for the LS and the homogeneity analysis.

Total Oxides of Nitrogen Concentration Value Assignment: The certified total oxides of nitrogen concentrations for this SRM lot were computed from the assigned concentration for the LS and the homogeneity analysis.

Stability: Periodic analyses of SRM units from this lot are performed at NIST to monitor stability. If significant changes in the nitric oxide concentration are observed, the purchaser will be notified.

REFERENCES

- [1] Taylor, B.N., "Guide for the Use of the International System of Units (SI)," NIST Special Publication 811, 1995 Ed., (April 1995).
- [2] Guide to the Expression of Uncertainty in Measurement, ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland, (1993): see also Taylor B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, U.S. Government Printing Office, Washington, DC (1994); (available at http://physics.nist.gov/Pubs/).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Telephone (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet http://ts.nist.gov/srm.